

# Certificate of Analysis<sup>(Ver.2.0)</sup>

## Deoxynivalenol

### 1. General information

This document is designed and the certified value(s) and uncertainty(ies) are determined in accordance with ISO Guide 31<sup>[1]</sup> and Eurachem / CITAC Guides<sup>[2,3]</sup>.

### 2. Description of the Reference Material (RM)

**Product name:** Deoxynivalenol

**Product number:** MSS1011

**CAS number:** 51481-10-8

**Formula:** C<sub>15</sub>H<sub>20</sub>O<sub>6</sub>

**Formula weight:** 296.32

**Lot#:** 2B0E25

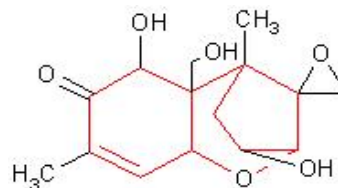
**Physical description of RM:** White crystalline powder of Deoxynivalenol

**Amount:** 1mg

**Production date:** 25,May,2022

**Expiry date:** 24,May,2025

**Name of the supplier:** Pribolab Pte. Ltd .  
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[www.pribolab.com](http://www.pribolab.com), [pribolab@gmail.com](mailto:pribolab@gmail.com)



#### 2.1 Intended use of the RM

- for laboratory use only
- calibration of analytical instruments

#### 2.2 Instruction for the correct use of the RM

The compound should be stored at -20°C in a dark place. Before usage of the RM, the compound should be allowed to warm to temperature (20±3°C). The recommended minimum sub-sample amount for all kinds of application is 1mg. Certified values and uncertainties can only be guaranteed if the minimum sampling volume requirement is met. The expiry date of this RM is based on the current knowledge and holds only for proper storage conditions in the originally closed flasks/ packages.

#### 2.3 Hazardous situation

The normal laboratory safety precautions should be observed when working with this RM. Further details for the handling of this RM are available as safety data sheet.

### 3. Certified values and their uncertainties

Deoxynivalenol		
Compound	Purity	
Deoxynivalenol	Certified value <sup>a</sup>	Uncertainty <sup>b</sup>
	99.5%	±0.5%
a The certified value is based upon the results from several analytical techniques		
b Expanded uncertainty U (k = 2) of the value uc according to GUM <sup>[4]</sup>		

### 4. Discussion of traceability

The qualitative analysis for principal component of the material is obtained by liquid chromatography-mass spectrometry (LC-MS), ultraviolet and visible spectroscopy (UV-VIS) and nuclear magnetic resonance (NMR). The certified value (purity of Deoxynivalenol) is based on the results of mass balance method and qNMR method. Structure related impurities are determined by liquid chromatography - tandem mass spectrometry (LC-MS/MS). Moisture content, inorganic impurities and volatile organic compounds are measured by Karl Fischer, ICP-MS and GC-FID respectively. Based on the above results, the purity certification value is given.

All weighting and dilution steps for preparation were done using calibrated equipment (microbalances, pipettes). The gravimetric preparation furthermore was performed in a standardized and certified class A flask with stated uncertainty as well as using traceable thermometers for temperature controlled preparation. The whole preparation process is therefore traceable to SI units and metrological traceability is given.

### 5. Purity assessment of Deoxynivalenol

#### 5.1 HPLC-DAD

The purity check using LC-DAD of the Deoxynivalenol sample showed one main peak after blank subtraction. The peak purity of the main signal was examined by diode array spectra of the Deoxynivalenol peak and led to the conclusion that this peak consists only of Deoxynivalenol.

column	C <sub>18</sub> , 250×4.6mm, 5μm	
injection Volume	50μL	
solvent	acetonitrile/water=10/90	
oven	35℃	
flow rate	1mL/min	
Sample dilution	methanol/water=50/50	
DAD settings	218nm	
	time[min]	area
Deoxynivalenol(DAD)	11.906	606960

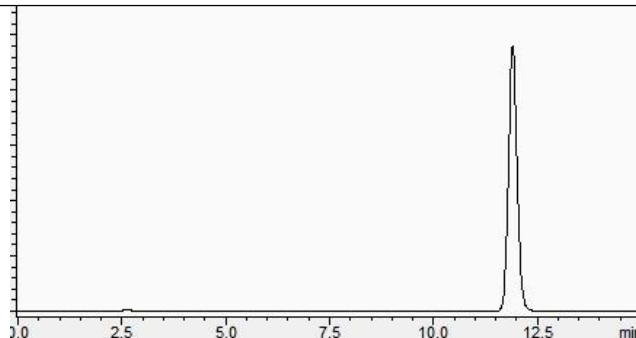


Figure 1: HPLC-DAD chromatogram of Deoxynivalenol .

### 5.2 LC-MS/MS

The purity check using gradient LC-MS/MS of the Deoxynivalenol sample showed one main peak after blank subtraction.

column	C18- column, 100*2.1mm, 3μm	
mobile phase		
solvent A:	water	
solvent B:	95%acetonitrile	
flow rate	0.3mL/min	
oven	30℃	
injection Volume	1μL	
gradient	time in minutes(min)	% solvent B
	0-2	20
	2-6	20-90
	6-8	90
	8-10	90-20
Source type	ESI, negative mode	

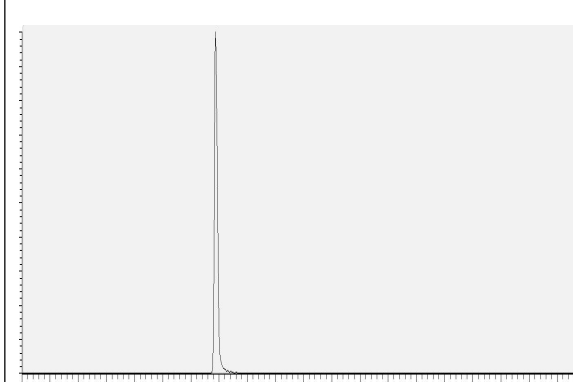


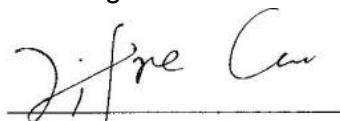
Figure 1: LC-MS/MS chromatogram of Deoxynivalenol

	time[min]	area
Deoxynivalenol	3.44	85692

## 6.Further information

The purchaser must determine the suitability of this product for its particular use. Pribolab makes no warranty of any kind, express or implied, other than its products meet all quality control standards set by Pribolab. We do not guarantee that the product can be used for a special application.

Inspected by



Quality System Specialist

## References:

- [1] ISO Guide 31:2015-1-18, "Reference materials—contents of certificates, labels and accompanying documentation"
- [2] Eurachem / CITAC Guide, 1-37, (2003), "Traceability in Chemical Measurement"
- [3] Eurachem / CITAC Guide CG4, 1-133, (QUAM:2012.P1), "Quantifying Uncertainty in Analytical Measurement", 3rd Ed.
- [4] International Organization for Standardization (ISO), (1995), "Guide to the Expression of Uncertainty in Measurement", 1<sup>st</sup> Ed. Geneva, Switzerland